



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>5</sup> :</b> <b>C11D 1/72, 17/00</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 92/20767</b> <b>(43) International Publication Date:</b> 26 November 1992 (26.11.92)
<b>(21) International Application Number:</b> PCT/US92/04162 <b>(22) International Filing Date:</b> 19 May 1992 (19.05.92) <b>(30) Priority data:</b> 702,180                      20 May 1991 (20.05.91)                      US <b>(71) Applicant:</b> DORSEY INDUSTRIES, INC. [— /US]; 227 Thorn Avenue, Orchard Park, NY 14127 (US). <b>(72) Inventor:</b> CONAWAY, Lawrence, Matthew ; 826 Pine Ave- nue, Niagara Falls, NY 14301 (US). <b>(74) Agent:</b> GREENWALD, Howard, J.; 330 Executive Office Building, 36 West Main Street, Rochester, NY 14614 (US).		<b>(81) Designated States:</b> AT, AT (European patent), BE (Euro- pean patent), CA, CH (European patent), DE (Euro- pean patent), DK (European patent), ES (European pa- tent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), KR, LU (Eu- ropean patent), MC (European patent), NL (European patent), SE (European patent).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the</i> <i>claims and to be republished in the event of the receipt of</i> <i>amendments.</i>
<b>(54) Title:</b> METHOD OF APPLYING INVERT DISPERSIONS OF NON-MISCIBLE SOLUTIONS  <b>(57) Abstract</b>  The process of the present invention is a method of applying invert dispersions of non-miscible solutions, comprising the steps of first selecting a first set of ingredients which will combine to form an active ingredient solution component and a second set of ingredients which will be combined to form a water solution component of the non-miscible solution; next, choosing an azeotrope of water which will reduce the evaporation point of water so that the water solution component evaporates along with of before the active ingredient solution component; then identifying the appropriate ratio of water to the azeotrope so as to cause the water solution component to evaporate at a constant composition at a desired temperature; and further, selecting a dispersal agent which will permit the active ingredient solution component to coexist with the water solution component and which will facilitate in creating an invert dispersion; then calculating the percent-by-weight composition of the first set of ingredients and the second set of ingredients; and then agitation the non-miscible solution prior to dispensing it in order to create a temporarily stable homogeneous solution; and finally dispensing the temporarily stable homogeneous solution.		

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Method of Applying Invert  
Dispersions of non Miscible Solutions

Technical Field

This invention relates to a process for dispensing a non-miscible water-based solution and specifically to dispensing a water-based cleanser onto a surface by the aerosol application of the cleanser as an invert dispersion.

Background Art

Aerosol systems are used for dispensing a wide variety of substances. Typically there are three components to any aerosol system: the active ingredient which is the substance being dispensed, the carrier which carries the active ingredient, and the propellant gas system which drives the active ingredient out of the aerosol can.

A commonly used propellant system is freon, which contains chloro-fluorocarbons (CFC's). CFC's are considered to be ozone depleters and because of their environmentally hazardous nature are becoming heavily regulated. In addition to propellant systems, many active ingredients and their carriers are environmentally dangerous or flammable, such as the universal cleanser 111 Trichlorethane.

Thus, there is a long felt need for an environmentally-friendly propellant system and cleanser. However, the challenge lies in the ability to replace as much as possible of the volatile and environmentally-friendly component such as water, but this must be done in such a combination so as to not diminish the cleansing ability of the cleanser ingredients.

One problem raised by this approach is that organic components are normally hydrophobic, i.e., they will not form an aqueous solution. Therefore, there is a need to

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find a way to blend the necessary organic cleansing components with water if an environmentally-safer cleanser is to be dispensed.

In addition, by introducing water into the cleanser dispensing system, there arises the threat that residual water will be left on the surface being cleansed which will in turn result in a pitting or corroding of that surface. So there also exists a need to assure that once the water-based cleanser is sprayed onto the surface, the water component will evaporate before, or along with, the organic cleansing component and not after.

#### Disclosure of Invention

The process of the present invention involves a method of applying invert dispersions of non-miscible solutions. In the first step of the process, there is selected a first set of ingredients which will combine to form an active ingredient solution component and a second set of ingredients which will be combined to form a water solution component of the non-miscible solution. Thereafter, there is chosen an azeotrope of water which will reduce the evaporation point of water so that the water solution component evaporates along with, or before, the active ingredient solution component. Then, the appropriate ratio of water to the azeotrope is identified so as to cause the water solution component to evaporate at a constant composition at a desired temperature. A dispersing agent is selected which will permit the active ingredient solution component to coexist with the water solution component and which will facilitate in creating an invert dispersion. The ingredients are then combined.

#### Brief Description of Drawings

Fig. 1 illustrates various embodiments of the water-based cleanser in terms of preferred percent by weight

composition of the components.

Fig. 2 illustrates the same embodiments as Fig. 1 but in terms of the preferred number of grams of each component.

#### Best Mode for Carrying Out the Invention

The present invention is a method for applying invert dispersions of non-miscible solutions, thereby allowing an active ingredient to be dispensed via a water-based carrier, as opposed to traditional, yet environmentally hazardous, organic aerosol carriers and without producing residual water deposits.

The process of the present invention comprises the following series of general steps.

The first step is to select a first set of ingredients and a second set of ingredients. The first set of ingredients will be combined to form an active ingredient solution component of a non-miscible solution and will be combined with the second set of ingredients, which make up a water solution component of the non-miscible solution. The active ingredient solution component and the water solution component combine to yield a solution whose active ingredient is dispensed via a water-based carrier.

The next step is to choose an azeotrope of water which will reduce the evaporation point of the water solution component below that of the active ingredient solution component. This can be done by consulting a table of boiling points of solvents and their azeotropic mixtures. After the azeotrope has been selected, the percent-by-weight composition of water in the solution will have to be determined with respect to the percent by weight of the azeotrope in order to cause the water solution component to evaporate at a constant composition at a requisite temperature. For example, in order to get water to evaporate at 1 atm at 73.5 degrees Celsius, as opposed to 100 degrees Celsius, a solution of water and its azeotrope, methyl ethyl ketone, must

be formed in a ratio of 11.6 percent by weight water to 88.4 percent methyl ethyl ketone. Any other combination will cause the azeotrope to boil off first.

The next step is to choose a dispersal agent. By producing a decrease in surface tension and an increase in electrical potential a dispersal agent permits an organic active ingredient solution component to coexist with the water solution component and facilitates in the formation of an invert dispersion. In an invert dispersion the natural formation of a droplet of a non-miscible solution is con-torted so that two solution components maintain positions opposite to that which would occur naturally. Once the appropriate azeotrope and dispersal agent have been identi-fied, it then becomes necessary to calculate the appropriate percent compositions of the ingredients of both the active ingredient solution component and the water solution compon-ent in order to assure the occurrence of an invert disper-sion and the reduction of the evaporation point of the water. The effectiveness of the surface active agent to-wards an invert dispersion is dependent on the proportion of water solution component to active ingredient solution component; therefore the limited and fixed portion of sur-face active agent must be calculated.

The formation of an invert dispersion requires a shift in the surface tension of the active ingredient solution component and the water solution component. To analytically confirm that such a shift will occur it is first necessary to measure the surface tension of each ingredient of the active ingredient solution component and of each ingredient of the water solution component.

Analytically, the surface tensions can be expressed by the following differential relation:  $dG = x dA$ , where  $dG$  is the differential surface tension value which the ingredient contributes to the solution;  $x$  is the surface tension of the ingredient in [dynes/cm]; and  $dA$  is the differential

area of the surface.

Once the surface tensions are known, the surface tension of the principle ingredient of the active ingredient solution component can be compared to that of the water solution component in order to determine an initial surface tension distribution ratio.

The addition of the Azeotrope and the dispersal agent causes a shift in the concentration as well as surface tension of the active ingredient solution component and the water solution component.

The new concentrations can be determined from the Nerst Distribution Law:  $K_d = C_2/C_1$ , where  $K_d$  is the distribution coefficient;  $C_2$  is the solute concentration 2; and  $C_1$  is the solute concentration 1.

The resulting surface tension shift reduces the surface tension of the water solution component below that of the active ingredient component. The shift occurs pursuant to J.W. Gibbs relation between Absorption and Interfacial Tension:  $Q = (-1/RT) \cdot (dx/d\ln a_2)$ , where  $Q$  is the surface area the surface excess of a solute species (in moles per square centimeter),  $a_2$  is the activity of the solute species and can be rewritten in terms of solute concentration as  $a_2 = c_2 z_2$ , where  $c_2$  is the solute concentration in moles per liter, and  $x_2$  is an activity coefficient whose value can be found in a table;  $R$  is the universal gas constant which has the value  $0.821.([L.atm]/[mol.K])$ ; and  $T$  is the temperature in degrees Kelvin.

By virtue of the shift in surface tension the water solution component occupies the core of a droplet and is encompassed by the active ingredient solution component. This being so, in an invert dispersion the water solution component takes on the persona of a solute while the active ingredient solution component acts as a solvent.

The new surface tension of the water solution component can be determined by virtue of the relation  $A = 1/QN_A$ ,

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where  $Q$  is expressed above in moles per square centimeter,  $N_A$  is avogadro's number which is  $6.02 \times 10^{23}$  moles<sup>-1</sup>;  $A$  is area in square centimeter; and the relation  $dG = x dA$ .

In order to onset an invert dispersion and create a temporary stable solution which will produce droplets of a constant homogeneous blend of a lyophobic solution when dispensed, it is necessary to agitate the solution is a caduceus lyophobic solution or short term suspension, agitation should not occur more than two minutes before dispensing. Beyond such time the solution is likely to separate back into its original two components.

As part of this process it is recommended that a temporary storage facility for the solution be selected as well as a means for dispensing the solution as droplets, such as an aerosol spray system. Typical delivery systems include aerosol or pump spray systems. However, in the case of bulk delivery an agitating tank and a pressurized sprayer may be required.

The steps of the process discussed above merely relate to the general application of this invention as a method of directly applying droplets of an active ingredient onto a surface using a water-based carrier so that in dispensing the active ingredient the environment will not be harmed and the surface will not be damaged by the carrier.

However, the preferred embodiment of this process has been tested and applied in the case of dispensing an organic cleanser onto a surface using an aerosol spray system which includes a propellant and a water-based carrier.

In this embodiment the first step of the process involves taking the ingredients of an aerosol cleanser and replacing as much as the environmentally-unfriendly and flammable ingredients with water but without diluting the effect of the cleanser. Here then the active ingredient is a cleansing ingredient, such as terpene, which as an organic is hydrophobic and so when combined with a water-based



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carrier forms a non-miscible solution. This non-miscible solution has two components; one, the active ingredient solution component, is comprised of a first set of ingredients which includes the organic cleanser and the propellant, while the other, the water solution component, is comprised of a second set of ingredients which includes water and the carrier. Glycol ether is the preferred carrier. The organic solution component has a greater density than the water solution component;

While a water-based carrier is more environmentally friendly than the typical organic aerosol carriers, the use of a water-based carrier may cause the surface in which the active ingredient is being dispensed onto to corrode. Therefore, in order to reduce the chance of corrosion it is preferred that the water solution component of the non-miscible cleansing solution be comprised of deionized water and rust inhibitors such as sodium nitrite and ammonium hydroxide.

Once the basic ingredients of this environmentally-friendly cleanser have been identified, the next step in the process is to select the appropriate azeotrope of water which will reduce the evaporation point of water below that of the cleanser so that the water will evaporate before the cleanser does. This prevents the formation of residual water deposits after the dispensed droplet has contacted the surface and separated back into the two components basic of the non-miscible solution. The preferred azeotrope is methyl ethyl ketone, and it insures that the water solution component will evaporate at a lower point than both pure water and the active ingredient solution component.

Next, a dispersal agent is selected. Here the preferred dispersal agent is monoethanolamine. Monoethanolamine is a mild surface active agent. In the preferred embodiment this chemical agent permits the otherwise hydrophobic organic active ingredient solution component to

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coexist temporarily with the water solution component as an invert dispersion. This occurs due to the fact that the dispersal agent shifts the surface tensions and the electrical potential of the droplet components.

After the appropriate azeotrope and dispersal agent are selected, the appropriate percent compositions of the ingredients of both the active ingredient solution component and the water solution component are determined so as to assure the occurrence of an invert dispersion and the reduction of the evaporation point of the water.

For the specific case of a cleansing solution the following percent compositions of the ingredients have been found to produce an invert dispersion of an azeotropic water solution. The water solution component is comprised of from about 60.0 to about 75.0 percent by weight water, from about 0.05 to about 0.20 percent by weight sodium nitrite, from about 0.10 to about 0.50 percent by weight ammonium hydroxide, from about 7.80 to about 9.20 percent by weight methyl ethyl ketone, from about 0.05 to about 0.10 percent by weight methyl formate, and from about 3.00 to about 5.00 percent by weight glycol ether. This combination of substances yields an environmentally-friendly carrier and does not diminish the capabilities of the active ingredient.

It is preferred that the water solution component be comprised of about 63.5 percent by weight water, about 0.14 percent by weight sodium nitrite, about 0.45 percent by weight ammonium hydroxide, about 7.83 percent by weight methyl ethyl ketone, about 0.09 percent by weight methyl formate, and about 3.90 percent by weight glycol ether.

The active ingredient solution component is comprised of from about 10.0 to about 20.0 percent by weight terpinenes, from about 0.75 to about 1.50 percent by weight monoethanolamine, and from about 7.00 to about 15.0 percent by weight propellant. The propellant contributes to the increase in density of the active ingredient solution component over the

water solution component.

It is preferred that the active ingredient solution component be comprised of about 13.4 percent by weight terpinenes, about 1.08 percent by weight monoethanolamine, and about 10.0 percent by weight propellant.

FIG. 1 appended to this specification further illustrates eight example compositions in approximate percent by weight of each component, and FIG. 2 similarly describes the same eight example compositions in approximate express weights. These compositions formulated by the method described above were found in laboratory trials to fully embody the functions and advantages of the methods of formulation and application described and claimed herein.

When the non-miscible cleansing solution of any of the above compositions is shaken prior to dispensing the formulation, a temporary stable homogeneous solution is formed such that when this solution is sprayed, bi-lateral droplets form. Due to the onset of an invert dispersion caused by the added dispersal agent, the water solution component is at the core of the droplet and is enveloped by active ingredient solution component.

In this specific application the overall result of this process is that an organic cleansing solution can be applied to a surface such that when dispensed as a spray of droplets, the cleanser will contact the surface before its water-based carrier, and the carrier will evaporate from the surface before the cleanser, thereby precluding the formation of residual water deposits on the surface which may have had a corrosive effect.

I claim:

1. A method of formulating invert dispersions of non-miscible solutions for application to a surface comprising the steps of:

(a) selecting a first set of ingredients which will combine an active ingredient phase and a second set of ingredients which will be combined to form a water carrier phase of the non-miscible solution;

(b) formulating said second set of ingredients by selecting an azeotrope of water which will reduce the evaporation point of water so that the water solution component will evaporate after application before the active ingredient solution component, identifying the appropriate ratio of water to the azeotrope so as to cause the water solution component to evaporate at a constant composition and at a desired temperature, and selecting a dispersal agent which will permit the active ingredient solution component to coexist with the water solution component and which will enable an invert dispersion when applied; and

(c) calculating the appropriate percent-by-weight composition of the first set of ingredients and the second set of ingredients to achieve a homogeneous invert dispersion and combining them in a container.

2. A method of applying an invert dispersion of a non-miscible solution as recited in claim 1 further comprising the steps of: agitating the non-miscible solution prior to dispensing it in order to create a temporarily stable homogeneous solution, and dispensing the temporarily stable homogeneous solution to said surface.

3. The method as recited in claim 1, wherein the step of selecting a first set of ingredients includes selecting a terpene in said first set of ingredients and the step of

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selecting the second set includes deionized water and glycol ether.

4. The method as recited in claim 1 wherein the step of choosing an azeotrope of water includes choosing methyl ethyl ketone.

5. The method as recited in claim 1, wherein the step of selecting a dispersal agent includes selecting monoethanolamine.

6. A method of applying invert dispersions of non-miscible solutions comprising the steps of:

(a) selecting a first set of ingredients which will combine to form an active ingredient solution component and a second set of ingredients which will be combined to form a water solution component of the non-miscible solution;

(b) formulating said second set by

1. choosing an azeotrope of water which will reduce the evaporation point of water so that the water solution component evaporates along with or before the active ingredient solution component;

2. identifying the appropriate ratio of water to the azeotrope so as to cause the water solution component to evaporate at a constant composition at a desired temperature; and

3. selecting a dispersal agent which will permit the active ingredient solution component to co-exist with the water solution component and which will facilitate in creating an invert dispersion;

(c) calculating the percent-by-weight composition of the first set of ingredients and the second set of ingredients and combining them in a container;

(d) agitating the non-miscible solution prior to dispensing it in order to create a temporarily stable homogeneous solution; and

(e) dispensing the temporarily stable homogeneous solu-

tion.

7. The method as recited in claim 6, wherein the step of selecting a first set of ingredients includes selecting a terpene in said first set of ingredients and the step of selecting the second set includes deionized water and glycol ether.
8. The method as recited in claim 6 wherein the step of choosing an azeotrope of water includes choosing methyl ethyl ketone.
9. The method as recited in claim 6 wherein the step of selecting a dispersal agent includes selecting monoethanolamine.
10. A non-miscible solution comprised of (a) an active ingredient, (b) a water-based carrier, (c) an azeotrope of water of a calculated proportion such that the evaporation point of the water-based carrier will be reduced below that of the active ingredient, (d) and a dispersal agent of a calculated proportion such that the surface tension of the water-based carrier is reduced so as to onset an invert dispersion.
11. The non-miscible solution as recited in claim 7, wherein the active ingredient is a terpene.
12. The non-miscible solution as recited in claim 7, wherein the water-based carrier phase includes glycol ether.
13. The non-miscible solution recited in claim 7, wherein the azeotrope of water is methyl ethyl ketone.
14. The non-miscible solution recited in claim 7, wherein the dispersal agent is monoethanolamine.
15. A non-miscible solution which after being agitated can be dispensed as an invert dispersion comprising:
  - (a) a water solution component, wherein said water solution component is comprised of from about 60.0 to about 75.0 percent by weight water, from about 0.05 to about 0.20 percent by weight sodium nitrite, from about 0.10 to

about 0.50 percent by weight ammonium hydroxide, from about 7.80 to about 9.20 percent by weight methyl ethyl ketone, from about 0.05 to about 0.10 percent by weight methyl formate, and from about 3.00 to about 5.00 percent by weight glycol ether;

(b) an active ingredient solution component, wherein said active ingredient solution component includes from about 10.0 to about 20.0 percent by weight terpinenes, from about 0.75 to about 1.50 percent by weight monoethanolamine, and from about 7.00 to about 15.0 percent by weight propellant.

16. The non-miscible solution recited in claim 12 wherein said water solution component comprises about 63.5 percent by weight water, about 0.14 percent by weight sodium nitrite, about 0.45 percent by weight ammonium hydroxide about 7.83 percent by weight methyl ethyl ketone, about 0.09 percent by weight methyl formate and about 3.90 percent by weight glycol ether, and wherein said active ingredient solution component comprises about 13.4 percent by weight terpinenes, about 1.08 percent by weight monoethanolamine, and about 10.0 percent by weight propellant.

17. A method of applying invert dispersions of non-miscible solutions comprising the steps of:

(a) selecting a first set of ingredients which will combine to form an active ingredient solution component and a second set of ingredients which will be combined to form a water solution component of the non-miscible solution;

(b) formulating said second set by selecting a dispersal agent which will permit the active ingredient solution component to coexist with the water solution component and which will facilitate in creating an invert dispersion;

(c) calculating the percent-by-weight composition of the

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first set of ingredients and the second set of ingredients and combining them in a container;

(d) agitating the non-miscible solution prior to dispensing it in order to create a temporarily stable homogeneous solution; and

(e) dispensing the temporarily stable homogeneous solution.

18. A non-miscible solution comprised of (a) an active ingredient; (b) a water-based carrier; (c) a dispersal agent of a calculated proportion such that the surface tension of the water-based carrier is reduced so as to onset an invert dispersion.



FIGURE 1

COMPOSITION IN PERCENT BY WEIGHT						
Percent By Weight						
1	2	3	4	5	6	
D.I. Water	71.80	69.50	69.50	71.40	67.80	
Methyl Ethyl Ketone	8.70	8.70	8.70	8.70	8.70	
Glycol Ether	4.00	4.00	4.00	4.00	4.00	
Sodium Nitrite	.15	.15	.15	.15	.15	
Terpine	14.90	14.90	14.90	14.90	14.90	
Monoethanolamine	.40	.40	.40	.40	.50	
Sorbitan 80 HLB <sup>4</sup>		2.40				
PMO 20 HLB <sup>14</sup>			2.40	.50		

SUBSTITUTE SHEET

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COMPOSITION IN GRAMS						
No. of Grams (Base Weight=200g)						
1	2	3	4	5	6	
D.I. Water	143.7	139.7	139.0	139.0	142.7	135.0
Methyl Ethyl Ketone	17.4	17.4	17.4	17.4	17.4	17.4
Glycol Ether	8.0	8.0	8.0	8.0	8.0	8.0
Sodium Nitrite	0.3	0.3	0.3	0.3	0.3	0.3
Terpine	29.8	29.8	29.8	29.8	29.8	29.8
Monoethanolamine	0.8	4.8	0.8	0.8	0.8	10.0
Sorbitan 80 HLB <sup>4</sup>			4.8			
PMO 20 HLB <sup>14</sup>				4.8	1.0	

FIGURE 2

SUBSTITUTE SHEET

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US92/04162

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : C11D 1/72, 17/00

US CL : 252/90, D169,548

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 252/90, D169,548 252/D169,90,105,153,170,171,305,548

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A, 5,102,573 (Han et al) 07 April 1992 See the Abstract	10-18
Y	US,A, 4,536,323 (Stopper) 20 August 1985 See the Abstract and Claim 1.	1-18
Y	US,A, 3,705,855 (Marschner) 12 December 1972 See Column 5.	1-18
Y	Mellon, <u>Industrial Solvents</u> , Reinhold Publishing Co. 2nd Edition, 1950 pg. 77	10-18

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

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Date of the actual completion of the international search

25 AUGUST 1992

Date of mailing of the international search report

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